

NASA TECH BRIEF

NASA Pasadena Office



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TLC Determination of Functionality in Prepolymers

The problem:

A rapid qualitative procedure was required to determine the functionality distribution in experimental prepolymers.

The solution:

Apply thin-layer chromatographic (TLC) techniques.

How it's done:

Commercially available glass plates coated with silica gel are used as the basis for the rapid determination of functionality distribution. The material to be analyzed is deposited as a small spot near one end of the plate; typically, a total of about 50 micrograms of solute is required (1 microliter of a 5% solution). When the spot is dry, the plate is placed, spot end down, in a shallow layer of a solvent contained in a tall container (the developing tank). The solvent rises on the silica layer on the plate by capillary action; as the solvent flows over the sample spot, the various components in the spot are eluted at different rates and tend to travel as a succession of spots along the thin silica film. After the solvent front has risen high enough (usually about 10 cm), its position is marked and the plate is removed from the developing tank, dried, and sprayed with a reagent which makes eluted material visible. Compounds are characterized by an R_f value, which is the fractional distance traveled by a given spot compared with the solvent front, both distances being measured from the point of application of the original spot.

TLC plates spotted with carboxyl-terminated ethylene/neohexene liquid copolymers are best developed by a 5-percent solution of ethanol in chloroform; 5-percent solutions of copolymer sample in

carbon tetrachloride are used for spotting. Effective reagents for visualization are aqueous potassium permanganate and a 5-percent solution of nitric acid in concentrated sulfuric acid.

Compounds with the highest functionality have the lowest R_f ; accordingly, difunctional fractions were found to have an R_f value between 0 and 0.4 (0 = starting point). Monofunctional fractions have R_f values between 0.5 and 0.8. Estimates of the relative amounts of n -functional fractions are made by comparison of sample spot sizes with those prepared from standard materials under identical conditions.

The functionality distribution is of fundamental importance for it determines the manner in which a given carboxyl-terminated prepolymer will cure and the physical properties of the resultant product. For example, a material containing equal amounts of non- and difunctional molecules would be expected to cure with a trifunctional aziridine or epoxide to give an insoluble gel plasticized with the nonfunctional component, whereas a material having the same average functionality of one carboxyl group per molecule but composed entirely of monofunctional molecules would not be expected to cure at all.

Note:

Requests for further information may be directed to:

Technology Utilization Officer
NASA Pasadena Office
4800 Oak Grove Drive
Pasadena, California 91103
Reference: TSP

(continued overleaf)

Patent status:

NASA has decided not to apply for a patent.

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